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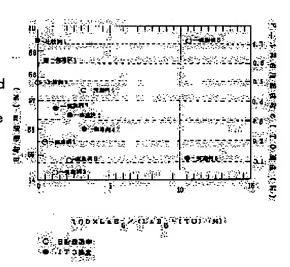
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(54) SUNLIGHT SHIELDING LAMINATED GLASS

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a sunlight shielding laminated glass improved in sunlight shielding function and having high transmissive performance to visible ray region. SOLUTION: The sunlight shielding laminated glass is formed by interposing an intermediate layer having the sunlight shielding

SOLUTION: The sunlight shielding laminated glass is formed by interposing an intermediate layer having the sunlight shielding function between 2 glass sheets. The intermediate layers is formed from an intermediate film composed of an adding liquid, which is prepared by dispersing at least one kind of hexaborate fine particle selected from a group composed of LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, LuB6, (La, Ce)B6, SrB6 and CaB6 and if necessary, ITO fine particle and/or ATO fine particle in a plasticizer, and a vinyl resin.



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CLAIMS

[Claim(s)]

[Claim 1] It is the solar radiation electric shielding glass laminate which makes the interlayer who has a solar radiation electric shielding function come to intervene between two sheet glass. Said interlayer LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, LuB6 (it La(s)), Ce) Glass laminate for solar radiation electric shielding characterized by being formed of the interlayer which consists of addition liquid which made the plasticizer distribute at least one sort of 6 boride particles chosen from the group which consists of B6, and SrB6 and CaB6, and vinyl system resin.

[Claim 2] It is the solar radiation electric shielding glass laminate which makes the interlayer who has a solar radiation electric shielding function come to intervene between two sheet glass. Said interlayer LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, LuB6 (it La(s)), Ce) Addition liquid which made the plasticizer distribute at least one sort of 6 boride particles chosen from the group which consists of B6, and SrB6 and CaB6 and an ITO particle, and/or an ATO particle, The glass laminate for solar radiation electric shielding characterized by being formed of the interlayer which consists of vinyl system resin.

[Claim 3] The glass laminate for solar radiation electric shielding according to claim 2 characterized by making the weight ratio of said 6 boride particle, and a said ITO particle and/or an ATO particle into the range of 0.1:99.9-90:10.

[Claim 4] The glass laminate for solar radiation electric shielding of claim 1-3 given in any 1 term with which said plasticizer is characterized by being triethylene glycol di-2-ethyl butyrate.

[Claim 5] It is the solar radiation electric shielding glass laminate which makes the interlayer who has a solar radiation electric shielding function come to intervene between two sheet glass. Said interlayer it forms in the field located inside one [at least] sheet glass -- having -- LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, and LuB6 (it La(s)), [and] Ce) The solar radiation screen which applied the coating liquid which contains at least one sort of 6 boride particles chosen from the group which consists of B6, and SrB6 and CaB6 as a solar radiation electric shielding component, and was formed, The glass laminate for solar radiation electric shielding characterized by being formed of the interlayer containing the vinyl system resin which intervenes between said two sheet glass.

[Claim 6] It is the solar radiation electric shielding glass laminate which makes the interlayer who has a solar radiation electric shielding function come to intervene between two sheet glass. Said interlayer it forms in the field located inside one [at least] sheet glass -- having -- LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, and LuB6 (it La(s)), [and] Ce) At least one sort of 6 boride particles chosen from the group which consists of B6, and SrB6 and CaB6, The glass laminate for solar radiation electric shielding characterized by being formed of the solar radiation screen which applied the coating liquid which contains one or more sorts in an ITO particle and an ATO particle as a solar radiation electric shielding component, and was formed, and the interlayer containing the vinyl system resin which intervenes between said two sheet glass.

[Claim 7] The glass laminate for solar radiation electric shielding according to claim 5 or 6 characterized by said interlayer having a solar radiation electric shielding function.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention] [0001]

[Field of the Invention] This invention relates to the glass laminate for solar radiation electric shielding used as the safety glass for cars, such as an automobile, a windowpane of a building, etc. [0002]

[Description of the Prior Art] The solar energy which puts a solar radiation screen and enters from glass between sheet glass as safety glass for [conventional] automobiles etc. is intercepted, and the thing aiming at mitigation of the feeling of **** of a cooling load or a man is proposed.

[0003] The solar energy which puts a solar radiation screen and enters from glass between sheet glass as safety glass for [conventional] automobiles etc. is intercepted, and the thing aiming at mitigation of the feeling of **** of a cooling load or a man is proposed. For example, according to JP,8-217500,A, this glass laminate makes the elasticity resin layer containing the heat ray electric shielding nature metallic oxide which consists of the tin oxide or indium oxide 0.1 micrometers or less of a detailed particle size intervene between the sheet glass of a pair, and is constituted. Moreover, the glass laminate which prepared and constituted the interlayer who distributed the metal of Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, Ce, In, nickel, Ag, Cu, Pt, Mn, Ta, W, V, and Mo, an oxide, a nitride, a sulfide, the dope objects of Sb or F, or these composites between at least two sheet glass is indicated by JP,8-259279,A. Furthermore, the windowpane for automobiles which made the glass component nature which consists of the ultrafine particle, the organic silicon, or organic silicon compound which consists of TiO2, ZrO2, SnO2, and In 2O2 intervene between transparence plate-like part material is indicated by JP.4-16004, A. To JP.10-297945, A further again between the transparence glass plate-like objects of at least two sheets The interlayer who consists of three layers is prepared. To the interlayer of the 2nd layer, among interlayers Sn, Ti, Si, Zn, Zr, Fe, aluminum, Cr, Co, In, nickel, Ag, The glass laminate which distributed the metal of Cu, Pt, Mn, Ta, W, V, and Mo, an oxide, a nitride, a sulfide, the dope objects of Sb or F, or these composites, and used the interlayer of the 1st layer and the 3rd layer as the resin layer is described. [0004]

[Problem(s) to be Solved by the Invention] However, the glass laminate concerning the Prior art indicated by these official reports had neither Japanese ***** nor enough light permeability ability, and the improvement was called for. This invention aims at offering the solar radiation electric shielding glass laminate which raises a solar radiation electric shielding function, and has the penetrable high ability of a light field. [0005]

[Means for Solving the Problem] The result to which this invention persons examined many things about the interlayer who consists of the interlayer or this interlayer which intervenes between two sheet glass, and a solar radiation screen in order to attain the above-mentioned purpose, This is ultrafine-particle-ized paying attention to 6 boride particle which holds a free electron so much. While producing the addition liquid which it comes to distribute to a plasticizer with ITO and an ATO particle according to a request, fabricating the vinyl system resin constituent which added this addition liquid to vinyl system resin in the shape of a sheet and forming an interlayer The glass of two sheets which applied the coating liquid which puts the interlayer formed in the shape of [said] a sheet between two sheet glass, or has a solar radiation electric shielding function inside, Or two sheet glass which applied one sheet glass and the coating liquid which has a solar radiation electric shielding function inside, and formed the solar radiation screen, A certain interlayer which it was and was formed as mentioned above between one sheet glass and other one usual sheet glass, Or by producing the glass laminate

for solar radiation electric sheding by the approach of putting the conventional interlayer which added the plasticizer to vinyl system resin and was fabricated in the shape of a sheet, and making it into an interlayer While this glass laminate for solar radiation electric shielding had the maximum of permeability in the light field, it finds out discovering absorption strong against a near infrared region, and having the minimum of permeability, and came to complete this invention.

[0006] Namely, the glass laminate for solar radiation electric shielding concerning the 1st embodiment of this invention It is the solar radiation electric shielding glass laminate which makes the interlayer who has a solar radiation electric shielding function come to intervene between two sheet glass. Said interlayer LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, LuB6 (it La(s)), Ce) It is characterized by being formed of the interlayer which consists of addition liquid which made the plasticizer distribute at least one sort of 6 boride particles chosen from the group which consists of B6, and SrB6 and CaB6, and vinyl system resin.

[0007] Moreover, the glass laminate for solar radiation electric shielding concerning the 2nd embodiment of this invention It is the solar radiation electric shielding glass laminate which makes the interlayer who has a solar radiation electric shielding function come to intervene between two sheet glass. Said interlayer LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, LuB6 (it La(s)), Ce) Addition liquid which made the plasticizer distribute at least one sort of 6 boride particles chosen from the group which consists of B6, and SrB6 and CaB6 and an ITO particle, and/or an ATO particle, It is characterized by being characterized by being formed of the interlayer which consists of vinyl system resin, and making the weight ratio of said 6 boride particle, and a said ITO particle and/or an ATO particle into the range of 0.1:99.9-90:10.

[0008] And in the 1st embodiment and 2nd embodiment of said this invention, said plasticizer is triethylene glycol di-2-ethyl butyrate.

[0009] The glass laminate for solar radiation electric shielding furthermore applied to the 3rd embodiment of this invention It is the solar radiation electric shielding glass laminate which makes the interlayer who has a solar radiation electric shielding function come to intervene between two sheet glass. Said interlayer it forms in the field located inside one [at least] sheet glass -- having -- LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, and LuB6 (it La(s)), [and] Ce) The solar radiation screen which applied the coating liquid which contains at least one sort of 6 boride particles chosen from the group which consists of B6, and SrB6 and CaB6 as a solar radiation electric shielding component, and was formed, It is characterized by being formed of the interlayer containing the vinyl system resin which intervenes between said two sheet glass.

[0010] The glass laminate for solar radiation electric shielding applied to the 4th embodiment of this invention further again It is the solar radiation electric shielding glass laminate which makes the interlayer who has a solar radiation electric shielding function come to intervene between two sheet glass. Said interlayer it forms in the field located inside one [at least] sheet glass -- having -- LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, and LuB6 (it La(s)), [and] Ce) At least one sort of 6 boride particles chosen from the group which consists of B6, and SrB6 and CaB6, It is characterized by being formed of the solar radiation screen which applied the coating liquid which contains one or more sorts in an ITO particle and an ATO particle as a solar radiation electric shielding component, and was formed, and the interlayer containing the vinyl system resin which intervenes between said two sheet glass.

[0011] And in the 3rd and 4th embodiments of said this invention, it is desirable that said interlayer has a solar radiation electric shielding function.
[0012]

[Embodiment of the Invention] In this invention, the following shall be first included as an interlayer's gestalt.

** The interlayer who consists only of an interlayer which consists of a vinyl system resin constituent which comes to add the addition liquid which made the plasticizer distribute a solar radiation electric shielding component to vinyl system resin while intervening between two sheet glass and sticking both sheet glass.

** The interlayer who consists of a solar radiation screen which applied the coating liquid which comes to add a binder in the addition liquid which was formed in the field located inside one [at least] sheet glass, and was distributed by the plasticizer in the solar radiation electric shielding component, and was formed, and an interlayer which consists of said vinyl system resin constituent which intervenes between two sheet glass and



sticks.

** The interlayer who consists of the conventional interlayer which comes to add a plasticizer to the vinyl system resin which intervenes between the solar radiation screen applied and formed and two sheet glass, and sticks the coating liquid which comes to add a binder to the addition liquid which was formed in the field located inside one [at least] sheet glass, and was distributed by the plasticizer in the solar radiation electric shielding component.

[0013] The addition liquid used for the solar radiation electric shielding glass laminate of this invention next distributes 6 boride particle, 6 boride particle, an ITO (tin content indium oxide) particle and 6 boride particle, the ATO (antimony content tin oxide) particle or 6 boride particle, ITO particle, and ATO particle as a solar radiation electric shielding component to homogeneity at the mixed liquor of a plasticizer and a solvent, and is produced.

[0014] and as a 6 boride particle used for this invention 6 hoe-ized lanthanum (LaB6), 6 hoe-ized cerium (CeB6), 6 hoe-ized praseodymium (PrB6), 6 hoe-ized neodymium (NdB6), 6 HOU-ized gadolinium (GdB6), 6 hoe-ized terbium (TbB6), 6 hoe-ized dysprosium (DyB6), 6 HOU-ized holmium (HoB6), 6 HOU-ized yttrium (YB6), 6 HOU-ized samarium (SmB6), 6 HOU-ized europium (EuB6), 6 hoe-ized erbium (ErB6), 6 HOU-ized thulium (TmB6), 6 hoe-ized ytterbium (YbB6), 6 hoe-ized lutetium (LuB6), The particle of 6 hoe-ized lanthanum cerium (La, Ce) (B6), 6 hoe-ized strontium (SrB6), and 6 HOU-ized calcium (CaB6) or the particle of such mixture is mentioned as the typical thing.

[0015] Moreover, although it is desirable that the front face has not oxidized as a 6 boride particle used for this invention, it is not avoided to some extent that have usually oxidized slightly in many cases, and surface oxidation takes place at the distributed process of a particle. However, the effectiveness which discovers a solar radiation shielding effect even in such a case is not affected at all.

[0016] Furthermore, although such a large solar radiation shielding effect that the integrity as a crystal is high is acquired, these 6 boride particles can discover a solar radiation shielding effect, if fundamental association inside a particle consists of association of each metal and boron, even if it seems that crystallinity produces a diffraction peak [low and broadcloth in an X diffraction].

[0017] Although light permeability arises in an interlayer or a solar radiation screen in the condition that particle size distributed in the interlayer or the solar radiation screen sufficiently small compared with light wavelength although these 6 boride particles were the powder colored ashes black, tea black, green black, etc., infrared light electric shielding ability can be held sufficiently strongly. Although it has not become clear in a detail, this reason has many amounts of the free electron in these particles, and since the absorbed energy of the indirect transition between bands by the free electron of the interior of a particle and a front face is just near visible - near-infrared, it is considered that the heat ray of this wavelength field is reflected and absorbed alternatively. By the film which distributed these particles to homogeneity finely enough according to the experiment, while permeability is the wavelength of 400nm - 700nm, it has the maximal value, and it has the minimal value with a wavelength of 700nm - 1800nm in between, and it is observed that the difference of the maximal value and the minimal value of these permeability is 15 points or more further. Light wavelength is 380nm - 780nm, if it takes into consideration that visibility is campanulate [with a peak of near 550nm], by such film, the light will be penetrated effectively, and the other heat ray will be reflected and absorbed effectively.

[0018] The ITO particle and ATO particle which are used next by this invention, combining with 6 boride particle have large reflection and absorption which reflection and absorption of light are not almost in a light field, and originates in plasma resonance in a field 1000nm or more. The lower right serves as ** as these transparency profiles go in a near infrared region to a long wavelength side.

[0019] On the other hand, the transparency profile of 6 borides has a bottom near 1000nm, and shows an upward slant to the right gradually by the long wavelength side from it. for this reason, by using 6 borides, and ITO and ATO together, light permeability becomes possible [covering the sunrays of a near infrared region], without making it decrease, and is independent respectively in an ITO particle or an ATO particle -- or a solar radiation electric shielding property can be raised rather than it combines and uses it.

[0020] Moreover, the solar radiation electric shielding capacity per unit weight of 6 borides is very high, and demonstrates the effectiveness by the 1/10 or less amount used as compared with ITO or ATO. By furthermore using together with ITO or ATO, since only a solar radiation electric shielding property can be raised further,

maintaining fixed light permeability, cost is also reducible. Moreover, since the amount of all the particles used is sharply reducible, it becomes possible to raise the abrasion strength and weatherability of an interlayer or a solar radiation screen.

[0021] Since a light field has absorption when 6 borides increase the amount used to a pan, by controlling the addition, absorption of a light field can be controlled freely and application of brightness adjustment, privacy protection, etc. can also be performed.

[0022] And as for the particle size of 6 boride particle to be used, it is desirable to be referred to as 200nm or less, and it sets it to 100nm or less more preferably. It is because a particle with larger mean particle diameter than 200nm or the condensed big and rough particle serves as a source of light scattering of the interlayer who consists of the fabricated interlayer or the applied solar radiation screen and this interlayer blooms cloudy. [0023] Moreover, an ITO particle and an ATO particle are also desirable and being referred to as 200nm or less by said same reason sets them to 100nm or less more preferably.

[0024] And if it is desirable that it is the range of 0.1:99.9-90:10 as for the weight ratio of 6 boride particle, and an ITO particle and/or an ATO particle and there are few 6 boride particles than this weight ratio range, the amount of all the particles used is seldom irreducible, the Kos ***** effectiveness will be small and a solar radiation electric shielding property will also worsen. On the other hand, even if there are more 6 boride particles than said weight ratio range, a solar radiation electric shielding property worsens.

[0025] Although the approach of distributing said particle to a solvent can be chosen as arbitration if it is an approach which a particle distributes in a solvent to homogeneity, as an example, approaches, such as a bead mill, a ball mill, a sand mill, and ultrasonic distribution, can be mentioned, and the addition liquid for an interlayer or solar radiation screens which uses said particle as a solar radiation electric shielding glass laminate of this invention by distributing to a solvent will be produced.

[0026] In addition, especially the solvent for adding said particle and distributing in said addition liquid, is not what is limited. It is possible to choose according to the vinyl system resin blended in case the conditions and vinyl system resin constituent which form an interlayer and a solar radiation screen are prepared. For example, dioctyl phthalate, dibutyl phthalate, diisobutyl phthalate, Adipic-acid-G 2-ethylhexyl, diisodecyl adipate, epoxy fatty acid monoester, Triethylene glycol di-2-ethyl butyrate, triethylene glycol-G 2-ethylhexoate, Plasticizers, such as a dibutyl sebacate and dibutyl sebacate, can be mentioned, and the various kinds of common organic solvents, such as alcohol, the ether, ester, and a ketone, are also usable. Moreover, an acid and alkali may be added if needed and pH may be adjusted.

[0027] In case [in which said vinyl system resin constituent is furthermore prepared] it hits and said particle is made to contain in vinyl system resin, in order to raise the distributed stability further, it is also possible to add the following plasticizers, various kinds of surfactants, a coupling agent, etc.

[0028] And it is not limited especially as a plasticizer added by said vinyl system resin, for example, dioctyl phthalate, dibutyl phthalate, diisobutyl phthalate, adipic-acid-G 2-ethylhexyl, diisodecyl adipate, epoxy fatty-acid-monoester triethylene glycol-G 2-ethyl butyrate, triethylene glycol-G 2-ethylhexoate, a dibutyl sebacate, dibutyl sebacate, etc. are mentioned.

[0029] As vinyl system resin used when preparing a vinyl system resin constituent using the addition liquid which is furthermore the above, and was made and prepared For example, a polyvinyl-butyral, polyvinyl chloride, and vinyl chloride-ethylene copolymer, A vinyl chloride-ethylene-glycidyl methacrylate copolymer, a vinyl chloride-glycidyl acrylate copolymer, A vinyl chloride-glycidyl methacrylate copolymer, a vinyl chloride-glycidyl acrylate copolymer, A polyvinylidene chloride and vinylidene-chloride-acrylonitrile copolymer, a polyvinyl acetate ethylene-vinylacetate copolymer, polyvinyl-acetal-polyvinyl-butyral mixture, etc. are mentioned. Especially a polyvinyl butyral is desirable.

[0030] A well-known approach is used for the formation approach of the interlayer which starts this invention next, for example, the calendering roll method, an extrusion method, the casting method, a tubular film process, etc. can be used. When using the interlayer which consists of a vinyl system resin constituent especially as the middle class for solar radiation electric shielding glass laminates of this invention, this vinyl system resin constituent can add and knead said addition liquid to vinyl system resin, and can fabricate the vinyl system resin constituent which a particle comes to distribute to homogeneity and was prepared in this way in the shape of a sheet. In case a vinyl system resin constituent is fabricated in the shape of a sheet, in order to blend a thermostabilizer, an antioxidant, etc. if needed and to raise the penetration of a sheet, an adhesive strength



regulator (for example, metal salt) may be blended.

[0031] To moreover, the field located inside [one / at least] said two sheet glass which constitutes the glass laminate for solar radiation electric shielding The coating liquid which makes binders, such as ethyl silicate, come to distribute 6 boride particle as a solar radiation electric shielding component is applied. Form a solar radiation screen or as a solar radiation electric shielding component 6 boride particle, Apply the coating liquid which makes said binder come to distribute one or more sorts in an ITO particle and an ATO particle, and a solar radiation screen is formed. Subsequently, among both sheet glass, the interlayer of the shape of a sheet which consists of the conventional interlayer which added the plasticizer to vinyl system resin and fabricated softening and the thing which liquefied in the shape of a sheet, or said vinyl system resin constituent is made to be able to intervene, it can be made to be able to stick, and the glass laminate for solar radiation electric shielding can also be formed.

[0032] Thus, according to this invention, the addition liquid which contains 6 boride particle as a solar radiation electric shielding component is added to vinyl system resin. The interlayer which fabricated the vinyl system resin constituent which the plasticizer was furthermore added, and homogeneity was distributed and was prepared in the shape of a sheet is used. Produce a glass laminate or Or apply the coating liquid which comes to add a binder in said addition liquid to the field located inside [one / at least] two sheet glass, and a solar radiation screen is formed. It becomes possible to offer the glass laminate for solar radiation electric shielding which has a solar radiation electric shielding function, without using the physical forming-membranes method and the complicated process of the high cost in producing a glass laminate using the interlayer or the conventional interlayer which consists of said vinyl system resin constituent, and has the penetrable high ability of a light region.

[0033] Moreover, 6 boride particle which has absorption strong against a near infrared region as a solar radiation electric shielding component, The interlayer which fabricated the vinyl system resin constituent which the addition liquid which used together the ATO particle and/or the ITO particle, and was prepared was added to vinyl system resin, the plasticizer was added further, and homogeneity was distributed, and was prepared in the shape of a sheet is used. Produce a glass laminate or Or apply the coating liquid which comes to add a binder in said addition liquid to the field located inside [one / at least] two sheet glass, and a solar radiation screen is formed. Or a solar radiation electric shielding property is raised rather than it combines and uses it. producing a glass laminate using the interlayer or the conventional interlayer which consists of said vinyl system resin constituent -- ATO and ITO -- independent in each particle -- The amount of the ATO particle at the time of production of an interlayer or a solar radiation screen or the ITO particle used is decreased, and it also becomes possible to reduce ingredient cost.

[0034]

[Example] The example of this invention is explained with the example of a comparison below. [0035] (Example 1) Optimum dose mixing of 20g [of LaB6 particles of 67nm of mean diameters] and diacetone alcohol (DAA) 50g, triethylene-glycol-di-2-ethyl-butyrate 20g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which LaB6 particle distributed was produced (A liquid).

[0036] On the other hand, optimum dose mixing of 20g [of ITO particles of 80nm of mean diameters] and triethylene-glycol-di-2-ethyl-butyrate 70g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which the ITO particle distributed was produced (B liquid).

[0037] The addition liquid for interlayers which mixed and prepared A liquid and B liquid was added to the polyvinyl butyral, triethylene glycol di-2-ethyl butyrate was added as a plasticizer, and 0.61% of the weight, ITO concentration prepared the vinyl system resin constituent so that 0.0038 % of the weight and polyvinyl-butyral concentration might become [LaB6 concentration] 70 % of the weight.

[0038] This vinyl system resin constituent was kneaded with a roll, it fabricated in the shape of [of 0.76mm thickness] a sheet, and the interlayer was produced. This interlayer was put between two transparent float glasses with a thickness of 2.5mm, after heating at 80 degrees C and carrying out temporary adhesion, 140 degrees C and a 14kg/cm2 autoclave performed this adhesion, and the glass laminate was produced. [0039] It measures using the Hitachi spectrophotometer and the spectral characteristic of the produced glass laminate is JIS. R 3106 is followed and they are solar radiation permeability and JIS. R Light permeability is



[0040] Moreover, as a result of measuring a pan mel value in the following way about the produced glass laminate, the pan mel value was 5 and the adhesion of sheet glass and an interlayer was enough. Pan mel trial: After keeping the glass laminate at -18 degrees C for 1 hour or more and making it constant temperature, it applied to the hammer (part of the head is 1 pound) grinding testing machine, and it ground until covering glass particle diameter was set to a maximum of 6mm or less. The broken piece of glass was shaken off and the part which the interlayer exposed was divided into the rank of 0-8, and it judged with a value becoming large, so that whenever [exposure] was low. It is the test method which judges whether this has the adhesion force of sheet glass and an interlayer within the limits of predetermined, and the value has desirable within the limits of 3-6. It is easy to exfoliate or less in one, and, on the other hand, penetration-proof becomes small or more by eight.

[0041] (Examples 2-5) The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about A liquid, B liquid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0042] (Example 6) The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about A liquid, the poly BIERU butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0043] (Example 1 of a comparison) The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about B liquid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0044] The presentation of the example 1 of a comparison and examples 1-5 and change of an optical property are shown in <u>drawing 1</u> below. It became possible from <u>drawing 1</u> to decrease solar radiation permeability further, without lowering light permeability compared with the property of the conventional ITO by carrying out minute amount addition of the LaB6 particle very much, and it turned out at this time that the amount of ITO particles is sharply reducible. For example, if the example 1 of a comparison is compared with an example 2, by having added LaB6 particle 1.38% of the weight to all the particles in an interlayer, light permeability can lower solar radiation permeability three points or more, maintaining to 78%, and can mitigate an ITO addition below in one half further. It was clearer than this that cost reduction can carry out to coincidence further with improvement in a solar radiation electric shielding property by minute amount addition of LaB6 particle. [0045] (Example 2 of a comparison) Optimum dose was mixed, ball mill mixing of 20g [of ATO particles of 55nm of mean diameters] and triethylene-glycol-di-2-ethyl-butyrate 70g, water, and the dispersant was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which the ATO particle distributed was produced (C fluid).

[0046] The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about C fluid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0047] (Examples 7-10) The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about A liquid, C fluid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0048] The presentation of the example 2 of a comparison and examples 7-10 and change of an optical property are shown in drawing 2 below. It became possible to decrease solar radiation permeability further, without lowering light permeability compared with the property of the conventional ATO by carrying out minute amount addition of the LaB6 particle very much from drawing 2. Moreover, it turned out at this time that the amount of ATO particles is sharply reducible. For example, when the example 2 of a comparison was compared with the example 8, maintaining light permeability to 78% by having added LaB6 particle 10.71% of the weight to all the particles in an interlayer, solar radiation permeability could be lowered about two points, and it turned out further that an ATO addition is reducible 60% or more.

[0049] (Example 11) Optimum dose mixing of 20g [of CeB6 particles of 85...m of mean diameters] and diacetone alcohol (DAA) 50g, triethylene-glycol-di-2-ethyl-butyrate 20g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which CeB6 particle distributed was produced (D liquid).

[0050] The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about D liquid, B liquid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0051] (Example 12) Optimum dose mixing of 20g [of PrB6 particles of 85nm of mean diameters] and diacetone alcohol (DAA) 50g, triethylene-glycol-di-2-ethyl-butyrate 20g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which PrB6 particle distributed was produced (E liquid).

[0052] The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about E liquid, B liquid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0053] (Example 13) Optimum dose mixing of 20g [of NdB6 particles of 85nm of mean diameters] and diacetone alcohol (DAA) 50g, triethylene-glycol-di-2-ethyl-butyrate 20g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which NdB6 particle distributed was produced (F liquid).

[0054] The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about F liquid, C fluid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0055] (Example 14) Optimum dose mixing of 20g [of GdB6 particles of 85nm of mean diameters] and diacetone alcohol (DAA) 50g, triethylene-glycol-di-2-ethyl-butyrate 20g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which GdB6 particle distributed was produced (G liquid).

[0056] The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about G liquid, C fluid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0057] (Example 15) Optimum dose mixing of 20g [of YB6 particles of 85nm of mean diameters] and diacetone alcohol (DAA) 50g, triethylene-glycol-di-2-ethyl-butyrate 20g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which YB6 particle distributed was produced (H liquid).

[0058] The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about H liquid, B liquid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0059] (Example 16) Optimum dose mixing of 20g [of SmB6 particles of 85nm of mean diameters] and diacetone alcohol (DAA) 50g, triethylene-glycol-di-2-ethyl-butyrate 20g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which SmB6 particle distributed was produced (I liquid).

[0060] The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about I liquid, C fluid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1.

[0061] (Example 17) Optimum dose mixing of 20g [of EuB6 particles of 85nm of mean diameters] and diacetone alcohol (DAA) 50g, triethylene-glycol-di-2-ethyl-butyrate 20g, water, and the dispersant was carried out, ball mill mixing was carried out for 100 hours using the zirconia ball with a diameter of 4mm, and 100g of addition liquid for interlayers which EuB6 particle distributed was produced (J liquid).

[0062] The vinyl system resin constituent was prepared so that it might become the presentation of Table 1 about J liquid, C fluid, a polyvinyl butyral, and triethylene glycol di-2-ethyl butyrate. The interlayer was produced for this by the same approach as an example 1, and the target glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 1. [0063]

[Table 1]

[Table I]		ピニル系	系 樹 脂 組 成 物		光学特性		
	6 赤	けん もず	ITO	ATO	ポリビニル	可視光	日射透
	雅 類	潰 度	漢皮	漁庆	プチラール	過率透	過率
		(%)	(%)	(%)	濃度(%)	(%)	(%)
英施例1	LaB ₆	0.0038	0. 61	0	70	78	55. 6
実施例2	LaB ₆	0.0051	0. 36	0	70	78	54. 2
実施例3	LaB6	0.0072	0. 32	0	70	78	54. 7
実施例4	LaB ₆	0.0086	0. 26	0	7 0	78	57. 5
実施例5	LaB ₆	0.014	0. 12	0	70	78	59. 3
実施例 6	LaB6	0.020	0	0	70	78	59. 7
比較例1	LaB ₆	0	0. 72	0	70	78	57.8
比較例2	LaB	0	0	0. 41	70	78	63. 1
实施例7	LaB6	0. 0038	0	0. 34	7 0	78	62. 5
実施例8	LaB6	0. 011	0	0. 18	70	78	61. 3
実施例9	LaB ₆	0.016	0	0. 13	70	78	61. 0
実施例10	LaB ₆	0.016	0	0. 099	70	78	62.0
実施例11	CeB ₆	0.0079	0. 33	0	70	77	53.8
実施例12	PrB ₆	0.0086	0. 32	0	70	77	54. 1
突旋例13	NdB ₆	0.0099	0	0. 18	70	78	62. 1
突施例14	GdB ₆	0. 011	0	0. 19	70	78	61. 9
卖施例15	YB 6	0. 0072	0. 29	0	70	78	54.0
实 施例16	SmB ₆	0.016	0	0. 13	7 0	77	61. 5
実施例17	EuB ₆	0. 0016	0	0. 12	70	77	61.8

[0064] (Example 18) The ethyl silicate solution which prepared the ethyl silicate 40 (the Tama chemical-industry incorporated company make: trade name) which is 4 - a pentamer in average degree of polymerization by 10g, ethanol 27g, 8g of 5% hydrochloric-acid water solutions, and 5g of water was mixed and agitated well, and 50g of ethyl silicate mixed liquor was prepared (K liquid).

[0065] A liquid, B liquid, and K liquid were mixed, and it diluted with diacetone alcohol further, and 7.25% of the weight, ITO concentration produced the coating liquid for solar radiation screens so that 0.045-% of the

weight and SiO2 concentration might become [LaB6 concentration] 2.5 % In the weight. 15g of this coating liquid was applied on the float glass with a thickness of 2.5mm by the spin coater, and it put into the 180-degree C electric furnace, and heated for 30 minutes, and the solar radiation electric shielding glass which made the solar radiation screen form on said float glass was produced.

[0066] This solar radiation electric shielding glass and a float glass with a thickness of 2.5mm were put by the 0.76mm polyvinyl-butyral film for interlayers, as said solar radiation screen faced inside, according to the usual glass laminate manufacturing method, heating and sticking by pressure of them were done, and the glass laminate was produced.

[0067] The spectral characteristic of the produced glass laminate is measured like an example 1, solar radiation permeability and light permeability are computed, and it is shown in Table 2. Moreover, as a result of carrying out the pan mel trial same about the produced glass laminate as an example 1 and measuring a pan mel value, the pan mel value was 5 and the adhesion of sheet glass and an interlayer was enough.

[0068] (Examples 19-22) A liquid, B liquid, and K liquid were mixed, and the coating liquid for solar radiation screens was produced so that it might dilute with diacetone alcohol further and might become the presentation of Table 2. Solar radiation electric shielding glass was produced by the same approach as an example 18 using this coating liquid, and the target glass laminate was produced using this. The optical property of this glass laminate is collectively shown in Table 2.

[0069] (Example 23) A liquid and K liquid were mixed, and the coating liquid for solar radiation screens was produced so that it might dilute with diacetone alcohol further and might become the presentation of Table 2. Solar radiation electric shielding glass was produced by the same approach as an example 18 using this coating liquid, and the target glass laminate was produced using this. The optical property of this glass laminate is collectively shown in Table 2.

[0070] (Example 24) A liquid, B liquid, and K liquid were mixed, and it diluted with diacetone alcohol further, and 3.63% of the weight, ITO concentration produced the coating liquid for solar radiation screens so that 0.023-% of the weight and SiO2 concentration might become [LaB6 concentration] 1.25% of the weight. Applied 15g of this coating liquid on the float glass with a thickness of 2.5mm by the spin coater, put into the 180-degree C electric furnace, heated for 30 minutes, the solar radiation screen was made to form on said float glass, and solar radiation electric shielding glass was produced.

[0071] On the other hand, in A liquid and B liquid, the polyvinyl butyral was mixed as vinyl system resin, triethylene glycol di-2-ethyl butyrate was mixed as a plasticizer, and 0.0025% of the weight, 0.18-% of the weight and LaB6 concentration prepared the vinyl system resin constituent so that polyvinyl-butyral concentration might become 70 % of the weight, and ITO concentration produced the interlayer fabricated by the same approach as an example 1.

[0072] And as said solar radiation screen faced said solar radiation electric shielding glass and a float glass with a thickness of 2.5mm inside, said interlayer was put, according to the usual glass laminate manufacturing method, heating and sticking by pressure of were done, and the glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 2.

[0073] As a result of measuring a pan mel value about the produced glass laminate, the pan mel value was 5 and the adhesion of sheet glass and an interlayer was enough.

[0074] (Example 25) A liquid and K liquid were mixed and it diluted with diacetone alcohol further, and 0.24% of the weight, LaB6 concentration produced the coating liquid for solar radiation screens so that SiO2 concentration might become 1.25 % of the weight. Applied 15g of this coating liquid on the float glass with a thickness of 2.5mm by the spin coater, put into the 180-degree C electric furnace, heated for 30 minutes, the solar radiation screen was made to form on said float glass, and solar radiation electric shielding glass was produced.

[0075] On the other hand, in A liquid and B liquid, the polyvinyl butyral was mixed as vinyl system resin, triethylene glycol di-2-ethyl butyrate was mixed as a plasticizer, and 0.0025% of the weight, 0.19-% of the weight and LaB6 concentration prepared the vinyl system resin constituent so that polyvinyl-butyral concentration might become 70 % of the weight, and ITO concentration produced the sheet-like interlayer fabricated by the same approach as an example 1.

[0076] And as said solar radiation screen faced inside with said solar radiation electric shielding glass and a float glass with a thickness of 2.5mm, said interlayer was put, according to the usual glass laminate

manufacturing method, heating and sticking by pressure of were done, and the glass laminate was produced. The optical property of this glass laminate is collectively shown in Table 2. [0077]

[Table 2]

}							
	È	全布衫	友の	組品	戈	光学	特性
		ウ 化 物	ITO	ATO	SiO2	可視光	日射透
ì	種類	濃 度	濃度	濃度	濃度	過率透	過率
		(%)	(%)	(%)	(%)	(%)	(%)
実施例18	LaB ₆	0. 045	7. 25	0	2. 5	78	55. 7
実施例19	LaB ₆	0.06	4. 33	0	2. 5	78	54. 1
実施例20	LaB ₆	0. 09	3. 84	0	2. 5	78	54. 5
実施例21	LaB ₆	0. 11	3. 09	0	2. 5	78	57. 7
実施例22	LaB ₆	0. 17	1. 38	0	2. 5	78	59. 4
実施例23	LaB ₆	0. 24	0	0	2. 5	78	59.6
実施例24	LaB ₆	0.023	3. 63	0	1, 25	78	58.0
実施例25	LaB ₆	0. 12	0	0	1. 25	7 8	57. 9

*実施例18~23は通常合わせガラスに用いられる中間膜を使用し、実施例24、25 は日射球磁成分を含有する中間膜を使用した。

[0078] In the glass laminate with which the interlayer for makeshift glass of two sheet glass intervenes as shown in each above example [whether the interlayer formed from the vinyl system resin constituent which adds the addition liquid which contains an ITO particle and/or an ATO particle according to 6 boride particle and a request to vinyl system resin, and is prepared by the simple approach is used without using the physical forming-membranes method of high cost, and] Or by applying the coating liquid which comes to add a binder in said addition liquid to the field which faces inside sheet glass, forming a solar radiation screen, and subsequently making said interlayer or conventional interlayer intervene It became possible to manufacture the solar radiation electric shielding glass laminate which has a solar radiation electric shielding function, and has penetrable high ability in a light region.

[Effect of the Invention] In the solar radiation electric shielding glass laminate which makes an interlayer come to intervene between two sheet glass as stated above according to this invention 6 boride particle and the interlayer formed from the vinyl system resin constituent which adds the addition liquid which contains an ITO particle and/or an ATO particle according to a request to vinyl system resin, and is prepared, After applying the coating liquid which comes to add said addition liquid to a binder to the field which faces inside sheet glass and forming a solar radiation screen, or by making said interlayer or conventional interlayer intervene It became possible to manufacture very easily the solar radiation electric shielding glass laminate which has a solar radiation electric shielding function, and has penetrable high ability in a light region by the simple approach, without using the physical forming-membranes method of high cost.

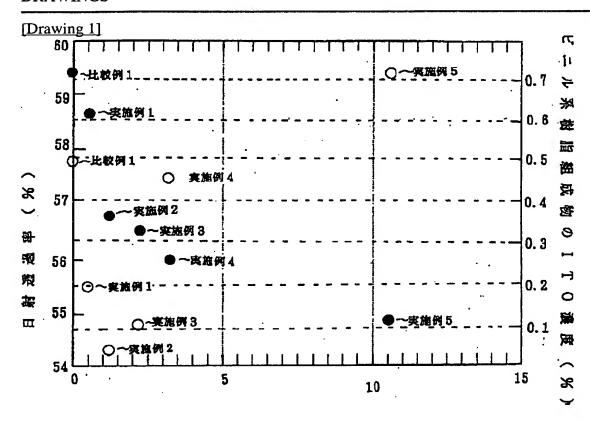
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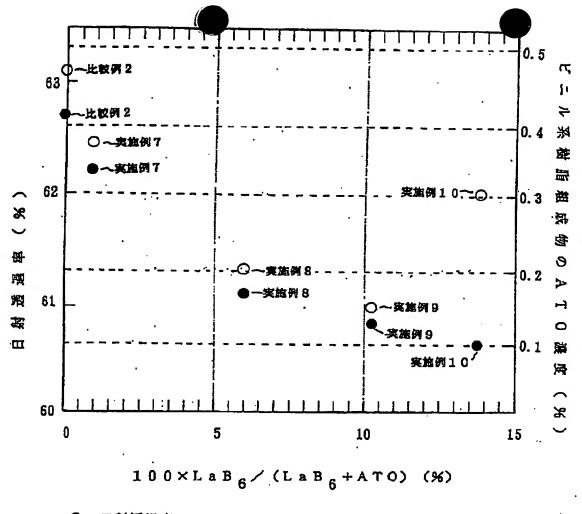
DRAWINGS



$$100 \times LaB_{6} / (LaB_{6} + ITO)$$
 (%)

- 〇 日射透過率
- ITO濃度

[Drawing 2]



- 〇 日射透過率
- ·ATO液度

[Translation done.]



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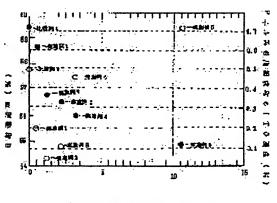
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(54) SUNLIGHT SHIELDING LAMINATED GLASS

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a sunlight shielding laminated glass improved in sunlight shielding function and having high transmissive performance to visible ray region.

SOLUTION: The sunlight shielding laminated glass is formed by interposing an intermediate layer having the sunlight shielding function between 2 glass sheets. The intermediate layers is formed from an intermediate film composed of an adding liquid, which is prepared by dispersing at least one kind of hexaborate fine particle selected from a group composed of LaB6, CeB6, PrB6, NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, YbB6, LuB6, (La, Ce)B6, SrB6 and CaB6 and if necessary, ITO fine particle and for ATO fine particle in a plasticizer, and a vinyl resin.



TODKLABEN (DEBELTO) /H

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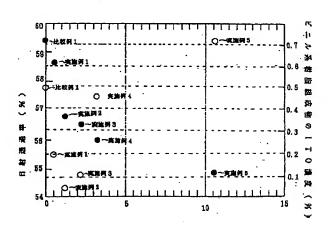
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(54) 【発明の名称】 日射遮蔽合わせガラス

(57)【要約】

日射遮蔽機能を高め、かつ可視光領域の高い 透過性能を有する日射遮蔽合わせガラスを提供する。

【解決手段】 日射遮蔽機能を有する中間層を2枚の板 ガラス間に介在せしめてなる日射遮蔽合わせガラスであ って、前記中間層は、LaB6、CeB6、PrB6、 NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, Y bB6、LuB6、(La, Ce) B6、SrB6およ びСаВ6からなる群から選択された少なくとも1種の 6ホウ化物微粒子、および所望に応じてITO微粒子お よび/またはATO微粒子とを可塑剤に分散させた添加 液と、ビニル系樹脂とからなる中間膜により形成される ことを特徴とする。



100×LaB6/(LaB6+1TO) (%)

- **年起进进**电

【特許請求の範囲】

日射遮蔽機能を有する中間層を2枚の板 【請求項1】 ガラス間に介在せしめてなる日射遮蔽合わせガラスであ って、前記中間層は、LaB₆、CeB₆、PrB₆、 NdB6, GdB6, TbB6, DyB6, HoB6, YB6, SmB6, EuB6, ErB6, TmB6, Y bB6、LuB6、(La、Ce) B6、SrB6およ びCaB6からなる群から選択された少なくとも1種の 6 ホウ化物微粒子を可塑剤に分散させた添加液と、ビニ ル系樹脂とからなる中間膜により形成されることを特徴 とする日射遮蔽用合わせガラス。

【請求項2】 日射遮蔽機能を有する中間層を2枚の板 ガラス間に介在せしめてなる日射遮蔽合わせガラスであ って、前記中間層は、LaBa、CeBa、PrBa、 NdB₆, GdB₆, TbB₆, DyB₆, HoB₆, YB6, SmB6, EuB6, ErB6, TmB6, Y bB6、LuB6、(La、Ce) B6、SrB6およ びСаВ6からなる群から選択された少なくとも1種の 6ホウ化物微粒子、ならびにITO微粒子および/また はATO微粒子とを可塑剤に分散させた添加液と、ビニ 20 ル系樹脂とからなる中間膜により形成されることを特徴 とする日射遮蔽用合わせガラス。

【請求項3】 前記6ホウ化物微粒子と、前記ITO微 粒子および/またはATO微粒子との重量比を 0.1: 99.9~90:10の範囲とすることを特徴とする請 求項2記載の日射遮蔽用合わせガラス。

【請求項4】 前記可塑剤が、トリエチレングリコール ジー2-エチルブチレートであることを特徴とする請 求項1~3のいずれか1項記載の日射遮蔽用合わせガラ ス。

【請求項5】 日射遮蔽機能を有する中間層を2枚の板 ガラス間に介在せしめてなる日射遮蔽合わせガラスであ って、前記中間層は、少なくとも一方の板ガラスの内側 に位置する面に形成され、かつLaB6、CeB6、P rB6, NdB6, GdB6, TbB6, DyB6, H oB6, YB6, SmB6, EuB6, ErB6, Tm B6, YbB6, LuB6, (La, Ce) B6, Sr B 6 および C a B 6 からなる群から選択された少なくと も1種の6ホウ化物微粒子を日射遮蔽成分として含有す る塗布液を塗布して形成された日射遮蔽膜と、前記2枚 40 の板ガラス間に介在されるビニル系樹脂を含有する中間 膜により形成されることを特徴とする日射遮蔽用合わせ ガラス。

【請求項6】 日射遮蔽機能を有する中間層を2枚の板 ガラス間に介在せしめてなる日射遮蔽合わせガラスであ って、前記中間層は、少なくとも一方の板ガラスの内側 に位置する面に形成され、かつLaB6、CeB6、P rBe, NdBe, GdBe, TbBe, DyBe, H oB6, YB6, SmB6, EuB6, ErB6, Tm B₆, Y_bB₆, L_uB₆, (La, Ce) B₆, S_r B6 およびCaB6 からなる群から選択された少なくと も1種の6ホウ化物微粒子と、ITO微粒子およびAT 〇微粒子のうち1種以上とを日射遮蔽成分として含有す る塗布液を塗布して形成された日射遮蔽膜と、前記2枚 の板ガラス間に介在されるビニル系樹脂を含有する中間 膜により形成されることを特徴とする日射遮蔽用合わせ ガラス。

【請求項7】 前記中間膜が日射遮蔽機能を有すること を特徴とする請求項5または6記載の日射遮蔽用合わせ ガラス。

【発明の詳細な説明】

[0001]

【発明の属する技術分野】本発明は、自動車などの車両 用の安全ガラス、建物の窓ガラスなどとして用いられる 日射遮蔽用合わせガラスに関するものである。

【従来の技術】従来の自動車用などの安全ガラスとして は、板ガラス間に日射遮蔽膜を挟み込んでガラスから入 る太陽エネルギーを遮断し、冷房負荷や人の熱暑感の軽 減を目的としたものが提案されている。

【0003】従来の自動車用などの安全ガラスとして は、板ガラス間に日射遮蔽膜を挟み込んでガラスから入 る太陽エネルギーを遮断し、冷房負荷や人の熱暑感の軽 減を目的としたものが提案されている。例えば特開平8 -217500号公報によれば、かかる合わせガラスは 一対の板ガラス間に、0.1 μm以下の微細な粒径の酸 化錫あるいは酸化インジウムからなる熱線遮蔽性金属酸 化物を含有した軟質樹脂層を介在せしめて構成されてい る。また特開平8-259279号公報には、少なくと も2枚の板ガラスの間に、Sn、Ti、Si、、Zn、 Zr, Fe, Al, Cr, Co, Ce, In, Ni, A g、Cu、Pt、Mn、Ta、W、V、Moの金属、酸 化物、窒化物、硫化物あるいはSbやFのドープ物また はこれらの複合物を分散した中間層を設けて構成した合 わせガラスが開示されている。さらに特開平4-160 04号公報にはTiO2、ZrO2、SnO2、In2 O2 からなる超微粒子と有機ケイ素あるいは有機ケイ素 化合物からなるガラス成分性とを透明板状部材の間に介 在させた自動車用窓ガラスが記載されている。さらにま た特開平10-297945号公報には少なくとも2枚 の透明ガラス板状体の間に、3層からなる中間層を設 け、中間層のうち第2層の中間層にはSn、Ti、S i, Zn. Zr, Fe, Al, Cr, Co, In, N i、Ag、Cu、Pt、Mn、Ta、W、V、Moの金 属、酸化物、窒化物、硫化物あるいはSbやFのドープ 物またはこれらの複合物を分散し、また第1層と第3層 の中間層を樹脂層とした合わせガラスを記述している。

[0004]

【発明が解決しようとする課題】しかしながら、これら 公報に記載された従来の技術に係る合わせガラスは日遮 機能や可視光透過性能が十分ではなく、その改善が求められていた。本発明は、日射遮蔽機能を高め、かつ可視 光領域の高い透過性能を有する日射遮蔽合わせガラスを 提供することを目的とするものである。

[0005]

【課題を解決するための手段】上記目的を達成するため に本発明者らは、2枚の板ガラスの間に介在する中間膜 または該中間膜と日射遮蔽膜からなる中間層について種 々検討した結果、自由電子を多量に保有する6ホウ化物 微粒子に着目し、これを超微粒子化し、所望に応じてⅠ TOやATO微粒子とともに可塑剤に分散してなる添加 液を作製し、この添加液をビニル系樹脂に添加したビニ ル系樹脂組成物をシート状に成形して中間膜を形成する とともに、2枚の板ガラス間に前記シート状に形成した 中間膜を挟み込むか、または日射遮蔽機能を有する塗布 液を内側に塗布した2枚のガラス、あるいは1枚の板ガ ラスと日射遮蔽機能を有する塗布液を内側に塗布して日 射遮蔽膜を形成した2枚の板ガラス、あるい1枚の板ガ ラスと他の通常の1枚の板ガラスの間に前記のように形 成した中間膜、もしくはピニル系樹脂に可塑剤を加えて シート状に成形した従来の中間膜を挟み込んで中間層と する方法で日射遮蔽用合わせガラスを作製することによ り、該日射遮蔽用合わせガラスが可視光領域に透過率の 極大を持つとともに、近赤外領域に強い吸収を発現して 透過率の極小を持つことを見出して本発明を完成するに 至った。

【0006】すなわち本発明の第1の実施態様に係る日射遮蔽用合わせガラスは、日射遮蔽機能を有する中間層を2枚の板ガラス間に介在せしめてなる日射遮蔽合わせガラスであって、前記中間層は、LaB6、CeB6、PrB6、NdB6、GdB6、TbB6、DyB6、HoB6、YB6、SmB6、EuB6、ErB6、TmB6、YbB6、LuB6、(La、Ce)B6、SrB6およびCaB6からなる群から選択された少なくとも1種の6ホウ化物微粒子を可塑剤に分散させた添加液と、ピニル系樹脂とからなる中間膜により形成されることを特徴とするものである。

 重量比を0.1:99.9~90:10の範囲とすることを特徴とするものである。

【0008】そして前記本発明の第1の実施態様および 第2の実施態様において、前記可塑剤が、トリエチレン グリコールージー2-エチルプチレートである。

【0011】そして前記本発明の第3および第4の実施 態様において、前記中間膜が日射遮蔽機能を有すること が好ましい。

[0012]

【発明の実施の形態】まず本発明においては中間層の形態としては以下を含むものとする。

①2枚の板ガラス間に介在して両板ガラスを密着するとともに、日射遮蔽成分を可塑剤に分散させた添加液をビニル系樹脂に添加してなるビニル系樹脂組成物からなる中間膜のみからなる中間層。

②少なくとも一方の板ガラスの内側に位置する面に形成され、かつ日射遮蔽成分を可塑剤に分散された添加液にパインダーを添加してなる塗布液を塗布して形成された日射遮蔽膜と、2枚の板ガラス間に介在して密着する前記ピニル系樹脂組成物からなる中間膜とからなる中間層。

③少なくとも一方の板ガラスの内側に位置する面に形成され、かつ日射遮蔽成分を可塑剤に分散された添加液にバインダーを添加してなる塗布液を塗布して形成された日射遮蔽膜と、2枚の板ガラス間に介在して密着するビニル系樹脂に可塑剤を加えてなる従来の中間膜とからなる中間層。

【0013】つぎに本発明の日射遮蔽合わせガラスに用いる添加液は、日射遮蔽成分としての6ホウ化物微粒子、6ホウ化物微粒子とITO(錫含有酸化インジウム)微粒子、6ホウ化物微粒子とATO(アンチモン含有酸化錫)微粒子、あるいは6ホウ化物微粒子とITO微粒子とATO微粒子を可塑剤と溶媒の混合液に均一に分散して作製されるものである。

【0014】そして本発明に使用される6ホウ化物微粒子としては、6ホウ化ランタン(LaBe)、6ホウ化セリウム(CeBe)、6ホウ化プラセオジム(PrBe)、6ホウ化ネオジム(NdBe)、6ホウ化ガドリニウム(GdBe)、6ホウ化テルピウム(TbBe)、6ホウ化ディスプロシウム(DyBe)、6ホウ化ホルミウム(HoBe)、6ホウ化イットリウム(YBe)、6ホウ化サマリウム(SmBe)、6ホウ化ユーロピウム(EuBe)、6ホウ化エルピウム(EuBe)、6ホウ化ルテチウム(LuBe)、6ホウ化リウム((La、Ce)Be)、6ホウ化ストロンチンセリウム(「Da、Ce)Be)、6ホウ化ストロンチの数粒子あるいはこれらの混合物の微粒子などが、その代表的なものとして挙げられる

【0015】また本発明に使用される6ホウ化物微粒子 30 としては、その表面が酸化していないことが好ましいが、通常は僅かに酸化していることが多く、また微粒子の分散工程で表面の酸化が起こることはある程度避けられない。しかしその場合でも日射遮蔽効果を発現する有効性には何ら影響を与えない。

【0016】さらにこれらの6ホウ化物微粒子は、結晶としての完全性が高いほど大きい日射遮蔽効果が得られるが、結晶性が低くX線回折でプロードな回折ピークを生じるようなものであっても、微粒子内部の基本的な結合が各金属とホウ素の結合からなり立っているものであるならば日射遮蔽効果を発現することができる。

【0017】これらの6ホウ化物微粒子は灰黒色、茶黒色、緑黒色などに着色した粉末であるが、粒径が可視光波長に比べて十分小さく中間膜中あるいは日射遮蔽膜中に分散した状態においては中間膜あるいは日射遮蔽膜に可視光透過性が生じるが、赤外光遮蔽能は十分強く保持できる。この理由は詳細には判明していないが、これら微粒子中の自由電子の量が多く、微粒子内部および表面の自由電子によるバンド間間接遷移の吸収エネルギーがまさに可視~近赤外の付近にあるために、この波長領域 50

の熱線が選択的に反射・吸収されるものと考えられる。 実験によればこれら微粒子を十分細かく、かつ均一に分 散した膜では、透過率が波長400nm~700nmの間に極大値を持ち、また波長700nm~1800nm の間に極小値を持ち、さらにこれらの透過率の極大値と 極小値の差が15ポイント以上であることが観察され る。可視光波長が380nm~780nmであり、視感 度が550nm付近をピークとする釣鐘型であることを 考慮すると、このような膜では可視光を有効に透過しそれ以外の熱線を有効に反射・吸収する。

【0018】つぎに本発明で6ホウ化物微粒子と併せて使用されるITO微粒子およびATO微粒子は、可視光領域で光の反射・吸収が殆どなく、1000nm以上の領域でプラズマ共鳴に由来する反射・吸収が大きい。これらの透過プロファイルは近赤外領域で長波長側に向かうにしたがって右下がりとなる。

【0019】一方、6ホウ化物の透過プロファイルは1000nm付近にボトムをもち、それより長波長側では徐々に右上がりを示す。このため6ホウ化物とITOやATOとを併用することによって可視光透過率は減少させずに、近赤外領域の太陽光線を遮蔽することが可能となり、ITO微粒子やATO微粒子をそれぞれ単独あるいは組合わせて使用するよりも日射遮蔽特性を向上させることができる。

【0020】また6ホウ化物の単位重量当たりの日射遮蔽能力は非常に高く、ITOやATOと比較して10分の1以下の使用量でその効果を発揮する。さらにITOやATOと併用することによって、一定の可視光透過率を保ちながら日射遮蔽特性のみを一層向上させることができるのでコストも削減できる。また全微粒子の使用量を大幅に削減することができるので、中間膜や日射遮蔽膜の摩耗強度や耐候性を向上させることが可能となる。

【0021】さらに6ホウ化物は使用量を増すと可視光 領域に吸収があるため、その添加量を制御することによ り可視光領域の吸収を自由に制御でき、明るさ調整やプ ライバシー保護などの応用もできる。

【0022】そして使用する6ホウ化物微粒子の粒径は200nm以下とすることが好ましく、より好ましくは100nm以下とする。平均粒径が200nmよりも大きい微粒子もしくは凝集した粗大粒子は、成形した中間膜または塗布した日射遮蔽膜からなる中間層の光散乱源となって該中間層が曇ってしまうからである。

【0023】またITO微粒子やATO微粒子も前記同様の理由で200nm以下とすることが好ましく、より好ましくは100nm以下とする。

【0024】そして6ホウ化物微粒子と、ITO微粒子および/またはATO微粒子との重量比は0.1:99.9~90:10の範囲であることが好ましく、この重量比範囲よりも6ホウ化物微粒子が少ないと、全微粒子の使用量をあまり削減できず、コスト削減効果が小さ

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く、日射遮蔽特性も悪くなる。一方前記重量比範囲より も6ホウ化物微粒子が多くても日射遮蔽特性が悪くなる。

【0025】前記微粒子を溶媒に分散する方法は、微粒子が均一に溶媒中に分散する方法であれば任意に選択できるが、例としてはビーズミル、ボールミル、サンドミル、超音波分散などの方法を挙げることができ、前記微粒子を溶媒に分散することによって本発明の日射遮蔽合わせガラスとして用いる中間膜あるいは日射遮蔽膜用添加液が作製されることになる。

【0026】なお前記添加液において前記微粒子を添加して分散するための溶媒は特に限定されるものではなく、中間膜や日射遮蔽膜を形成する条件やビニル系樹脂 は成物を調製する際に配合されるビニル系樹脂などに合わせて選択することが可能であり、例えばジオクチルフタレート、ジブチルフタレート、ジイソブチルフタレート、ジイソデシルである。エたアルコール・ジブチルカリュール・ジークリコール・ジークリコール・ジークリコール・ジークリコール・ジークリコール・ジークリコール・ジークリコール・ジークリコール・ジークリコール・セバケートなどの可塑剤を挙げることができ、またアルコール、エーテル、ケトンなどの一般的な有機溶媒の各種も使用可能である。また必要に応じて酸やアルカリを添加してpHを調整してもよい。

【0027】さらに前記ビニル系樹脂組成物を調製する当たり、ビニル系樹脂中に前記微粒子を含有させる際にその分散安定性を一層向上させるために、以下の可塑剤や各種の界面活性剤、カップリング剤などを添加することも可能である。

【0028】そして前記ピニル系樹脂に添加される可塑剤としては特に限定されず、例えばジオクチルフタレート、ジプチルフタレート、ジイソプチルフタレート、アジピン酸ージー2ーエチルヘキシル、アジピン酸ジイソデシル、エポキシ脂肪酸モノエステルトリエチレングリコールージー2ーエチルブチレート、トリエチレングリコールージー2ーエチルペキソエート、セバシン酸ジプチル、ジプチルセバケートなどが挙げられる。

【0029】さらに前記のようにして調製された添加液を用いてビニル系樹脂組成物を調製する場合に使用されるビニル系樹脂としては、例えばポリビニルブチラール、ボリ塩化ビニル、塩化ビニルーエチレン共重合体、塩化ビニルーエチレンーグリシジルメタクリレート共重合体、塩化ビニルーエチレンーグリシジルメタクリレート共重合体、塩化ビニルーグリシジルメタクリレート共重合体、塩化ビニルーグリシジルメタクリレート共重合体、塩化ビニルーグリシジルアクリレート共重合体、ポリ塩化ビニリデン、塩化ビニリデンーアクリロニトリル共重合体、ポリ酢酸ビニルエチレンー酢酸ビニル共重合体、ポリビニルアセタールーポリビニルブチラール混合物などが挙げられる。特にポリビニルブチラールが好50

ましい。

【0030】つぎに本発明に係る中間膜の形成方法には公知の方法が用いられ、例えばカレンダーロール法、押出法、キャスティング法、インフレーション法などを用いることができる。特に本発明の日射遮蔽合わせガラス用中間層としてビニル系樹脂組成物からなる中間膜を用いる場合、該ビニル系樹脂組成物はビニル系樹脂に前記添加液を添加して、混練して微粒子が均一に分散してなるものであり、このように調製されたビニル系樹脂組成物をシート状に成形することができる。ビニル系樹脂組成物をシート状に成形する際には、必要に応じて熱安定剤、酸化防止剤などを配合し、またシートの貫通性を高めるために接着力調整剤(例えば金属塩)を配合してもよい。

【0031】また日射遮蔽用合わせガラスを構成する前記2枚の板ガラスの少なくとも一方の内側に位置する面に、日射遮蔽成分として6ホウ化物微粒子をエチルシリケートなどのバインダーに分散させてなる塗布液を塗布して日射遮蔽膜を形成したり、あるいは日射遮蔽成分として6ホウ化物微粒子と、ITO微粒子およびATO微粒子のうち1種以上とを前記バインダーに分散させてる塗布液を塗布して日射遮蔽膜を形成し、ついで両板ガラス間にピニル系樹脂に可塑剤を添加して軟化・液状化したものをシート状に成形した従来の中間膜あるいは前記ピニル系樹脂組成物からなるシート状の中間膜を介在せしめて密着させて日射遮蔽用合わせガラスを形成することもできる。

【0032】このように本発明によれば、日射遮蔽成分として6ホウ化物微粒子を含有する添加液をビニル系樹脂に添加し、さらに可塑剤を加えて均一に分散させて調製されたビニル系樹脂組成物をシート状に成形した中間膜を用いて合わせガラスを作製したり、または前記添加液にバインダーを添加してなる塗布液を2枚の板ガラスの少なくとも一方の内側に位置する面に塗布して日射遮蔽膜を形成し、前記ビニル系樹脂組成物からなる中間膜あるいは従来の中間膜を用いて合わせガラスを作製することで高コストの物理成膜法や複雑な工程を用いずに日射遮蔽機能を有し、かつ可視光域の高い透過性能を有する日射遮蔽用合わせガラスを提供することが可能となる。

【0033】また日射遮蔽成分として、近赤外領域に強い吸収を持つ6ホウ化物微粒子と、ATO微粒子および/またはITO微粒子とを併用して調製された添加液をビニル系樹脂に添加しさらに可塑剤を加えて均一に分散させて調製されたビニル系樹脂組成物をシート状に成形した中間膜を用いて合わせガラスを作製したり、または前記添加液にパインダーを添加してなる塗布液を2枚の板ガラスの少なくとも一方の内側に位置する面に塗布して日射遮蔽膜を形成し、前記ビニル系樹脂組成物からなる中間膜あるいは従来の中間膜を用いて合わせガラスを

作製することでATOやITOそれぞれの微粒子を単独 あるいは組合わせて使用するよりも日射遮蔽特性を向上 させ、中間膜や日射遮蔽膜の作製時のATO微粒子やI TO微粒子の使用量を減少させ、材料コストを低減させ

[0034]

ることも可能となる。

【実施例】以下に本発明の実施例を比較例とともに説明 する。

【0035】(実施例1)平均粒径67nmのLaB6 微粒子20g、ジアセトンアルコール(DAA)50g、トリエチレングリコールージー2-エチルブチレート20g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、LaB6微粒子が分散した中間膜用添加液100gを作製した(A液)。

【0036】一方平均粒径80nmのITO微粒子20g、トリエチレングリコールージー2-エチルプチレート70g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、ITO微粒子が分散した中間膜用添加液100gを20作製した(B液)。

【0037】A液とB液を混合して調製した中間膜用添加液をポリビニルブチラールに添加し、可塑剤としてトリエチレングリコールージー2ーエチルブチレートを加え、ITO濃度が0.61重量%、LaB6濃度が0.0038重量%、ポリビニルブチラール濃度が70重量%となるようにビニル系樹脂組成物を調製した。

【0038】このピニル系樹脂組成物をロールで混練して0.76mm厚のシート状に成形して中間膜を作製した。この中間膜を厚さ2.5mmの透明なフロートガラ 30 \mathbb{Z} ス2枚の間に挟み込み、80℃に加熱して仮接着した後、140℃、14kg/cm²のオートクレーブにより本接着を行い、合わせガラスを作製した。

【0039】作製された合わせガラスの分光特性は日立製作所製の分光光度計を用いて測定し、JIS R 3106にしたがって日射透過率、またJIS R 3211にしたがって可視光透過率を算出し、その結果を表1に示す。

【0040】また作製された合わせガラスについて下記の要領でパンメル値を測定した結果、パンメル値は5で 40 あり、板ガラスと中間膜との密着性は十分であった。パンメル試験:合わせガラスを1時間以上−18℃に保って恒温にした後、ハンマー(頭の部分が1ポンドである)粉砕試験機にかけ、被着ガラス粒子径が最大6mm以下になるまで粉砕した。割れたガラス片を振り落とし、中間膜の露出した部分を0~8のランクに分けて、露出度が低いほど値が大きくなるように判定した。これは板ガラスと中間膜との密着力が所定の範囲内にあるかどうかを判定する試験方法であり、その値は3~6の範囲内が望ましい。1以下では剥離し易く、一方8以上で 50

は耐貫通性が小さくなる。

【0041】(実施例 $2\sim5$) A液、B液、ポリビニルプチラール、トリエチレングリコールージー<math>2-エチルプチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0042】(実施例6)A液、ポリビエルブチラール、トリエチレングリコールージー2ーエチルブチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0043】(比較例1) B液、ポリビニルブチラール、トリエチレングリコールージー2ーエチルブチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0044】つぎに比較例1および実施例1~5の組成と光学特性の変化を図1に示す。図1よりLaB6微粒子をごく微量添加することで、従来のITOの特性に比べて可視光透過率を下げずに、さらに日射透過率を減少させることが可能となり、またこの時ITO微粒子量を大幅に削減できることが分った。例えば比較例1と実施例2とを比較すると、LaB6微粒子を中間膜中の全微粒子に対して1.38重量%添加したことによって、可視光透過率は78%に維持したまま日射透過率を3ポイント以上下げることができ、さらにITO添加量を半分以下に軽減できる。これよりLaB6微粒子の微量添加で日射遮蔽特性の向上と、さらにコスト削減とが同時に行なえることが明らかであった。

【0045】(比較例2)平均粒径55nmのATO微粒子20g、トリエチレングリコールージー2-エチルプチレート70g、水および分散剤を適量を混合して直径4mmのジルコニアポールを用いて100時間ボールミル混合して、ATO微粒子が分散した中間膜用添加液100gを作製した(C液)。

【0046】C液、ポリビニルブチラール、トリエチレングリコールージー2ーエチルブチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0047】(実施例7~10)A液、C液、ポリビニルプチラール、トリエチレングリコールージー2-エチルプチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0048】つぎに比較例2および実施例7~10の組成と光学特性の変化を図2に示す。図2よりLaB6微粒子をごく微量添加していくことで、従来のATOの特性に比べて可視光透過率を下げずに、さらに日射透過率を減少させることが可能となった。またこの時ATO微粒子量を大幅に削減できることが分った。例えば比較例2と実施例8とを比較すると、LaB6微粒子を中間膜中の全微粒子に対して10.71重量%添加したことによって、可視光透過率は78%に維持したまま、日射透過率を2ポイント程度下げることができ、さらにATO添加量を60%以上削減できることが分った。

【0049】(実施例11) 平均粒径85nmのCeB6微粒子20g、ジアセトンアルコール(DAA)50g、トリエチレングリコールージー2ーエチルブチレート20g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、CeB6微粒子が分散した中間膜用添加液100gを作製した(D液)。

【0050】D液、B液、ポリビニルブチラール、トリエチレングリコールージー2ーエチルブチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0051】(実施例12) 平均粒径85nmのPrB 6 微粒子20g、ジアセトンアルコール(DAA) 50g、トリエチレングリコールージー2-エチルブチレート20g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、PrB6 微粒子が分散した中間膜用添加液100gを作製した(E液)。

【0052】 E液、B液、ポリビニルブチラール、トリエチレングリコールージー2 - エチルブチレートを表 1 の組成になるようにビニル系樹脂組成物を調製した。これを実施例 1 と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表 1 に併せて示す。

【0053】(実施例13) 平均粒径85nmのNdB 6 微粒子20g、ジアセトンアルコール(DAA) 50 g、トリエチレングリコールージー2-エチルプチレー 40ト20g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、NdB6 微粒子が分散した中間膜用添加液100gを作製した(F液)。

【0054】 F液、C液、ポリビニルブチラール、トリエチレングリコールージー2-エチルブチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0055】(実施例14) 平均粒径85nmのGdB6微粒子20g、ジアセトンアルコール(DAA)50g、トリエチレングリコールージー2-エチルブチレート20g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、GdB6微粒子が分散した中間膜用添加液100gを作製した(G液)。

【0056】G液、C液、ポリピニルブチラール、トリエチレングリコールージー2-エチルブチレートを表1の組成になるようにピニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0057】(実施例15)平均粒径85nmのYB6 微粒子20g、ジアセトンアルコール(DAA)50g、トリエチレングリコールージー2ーエチルブチレート20g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、YB6 微粒子が分散した中間膜用添加液100gを作製した(H液)。

【0058】 H液、B液、ポリビニルブチラール、トリエチレングリコールージー2ーエチルブチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0059】 (実施例16) 平均粒径85nmのSmB 6 微粒子20g、ジアセトンアルコール (DAA) 50g、トリエチレングリコールージー2ーエチルブチレート20g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、SmB 6 微粒子が分散した中間膜用添加液100gを作製した(I液)。

【0060】 I 液、C液、ポリビニルブチラール、トリエチレングリコールージー2ーエチルブチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とする合わせガラスを作製した。この合わせガラスの光学特性を表1に併せて示す。

【0061】 (実施例17) 平均粒径85nmのEuB 6 微粒子20g、ジアセトンアルコール (DAA) 50g、トリエチレングリコールージー2ーエチルプチレート20g、水および分散剤を適量混合し、直径4mmのジルコニアボールを用いて100時間ボールミル混合して、EuB 6 微粒子が分散した中間膜用添加液100gを作製した(J液)。

【0062】 J液、C液、ポリビニルブチラール、トリエチレングリコールージー2ーエチルブチレートを表1の組成になるようにビニル系樹脂組成物を調製した。これを実施例1と同様の方法で中間膜を作製して目的とす



る合わせガラスを作製した。この合わせガラスの光学特

[0063]

性を表1に併せて示す。

【表1】

	ピニル系		樹脂組成物					
1		ア 化 物	OTI	ATO	ポリピニル	可視光	日射透	
1	種類	潰 庻	漢度	独皮	プチラール	過率透	通率	
		(%)	(%)	(%)	渡皮(%)	(%)	(%)	
実施例1	LaB ₆	0.0038	0. 61	0	7 0	78	55. 6	
実施例2	LaB ₆	0.0051	0. 36	0	70	7 8	54. 2	
実施例3	LaB ₆	0.0072	0. 32	0	7 0	78	54. 7	
実施例4	LaB ₆	0.0086	0. 26	0	70	78	57. 5	
実施例5	LaB ₆	0.014	0. 12	0	70	78	59. 3	
実施例 6	LaB ₆	0.020	0	0	7 0	78	59. 7	
比較例1	LaB ₆	. 0 .	0. 72	0	70	78	57. B	
比較例2	LaB ₆	0	0	0. 41	70	78	63. 1	
実施例7	LaB ₆	0. 0038	0	0. 34	70	78	62. 5	
実施例8	LaB ₆	0. 011	0	0. 18	7 0	78	61. 3	
実施例 9	LaB ₆	0.016	0 .	0. 13	7 0	78	61. 0	
実施例10	LaB ₆	0.016	0	0. 099	7 0	78	62.0	
実施例11	CeB ₆	0.0079	0. 33	0	7 0	77	53.8	
実施例12	PrB ₆	0.0086	0. 32	0	7 0	77	54. 1	
実施例13	NdB ₆	0. 0099	0	0. 18	7 0	78	62. 1	
実施例14	GdB ₆	0.011	0	0. 19	70	78	61. 9	
実施例15	Y B 6	0.0072	0. 29	0	7 0	78	54.0	
奥施例16	SmB ₆	0.016	0	0. 13	70	77	61.5	
実施例17	EuB ₆	0.0016	0	0. 12	7 0	77	61. 8	

【0064】 (実施例18) 平均重合度で4~5量体で 40 あるエチルシリケート40(多摩化学工業株式会社製: 商品名)を10g、エタノール27g、5%塩酸水溶液 8g、水5gで調製したエチルシリケート溶液をよく混 合・撹拌してエチルシリケート混合液50gを調製した (K液)。

【0065】A液とB液とK液を混合し、さらにジアセ トンアルコールで希釈して、ITO濃度が7.25重量 %、LaB6 濃度が0.045 重量%、SiO2 濃度が 2. 5重量%となるように日射遮蔽膜用塗布液を作製し た。この塗布被15gをスピンコーターで厚さ2.5m so 率を算出し、表2に示す。また作製された合わせガラス

mのフロートガラス上に塗布して180℃の電気炉に入 れて30分間加熱し、前記フロートガラス上に日射遮蔽 膜を形成させた日射遮蔽ガラスを作製した。

【0066】この日射遮蔽ガラスと厚さ2.5mmのフ ロートガラスとを前記日射遮蔽膜が内側に面するように して0.76mmの中間膜用ポリピニルブチラール膜で 挟み込んで通常の合わせガラス製造法にしたがって加熱 ・圧着して合わせガラスを作製した。

【0067】作製された合わせガラスの分光特性を実施 例1と同様にして測定し、日射透過率および可視光透過



について実施例1同様のパンメル試験を実施してパンメル値を測定した結果、パンメル値は5であり、板ガラスと中間膜との密着性は十分であった。

【0068】(実施例19~22) A液とB液とK液を混合し、さらにジアセトンアルコールで希釈して表2の組成になるように日射遮蔽膜用塗布液を作製した。この塗布液を用いて実施例18と同様の方法で日射遮蔽ガラスを作製しこれを用いて目的とする合わせガラスを作製した。この合わせガラスの光学特性を表2に併せて示す。

【0069】(実施例23)A液とK液を混合し、さらにジアセトンアルコールで希釈して表2の組成になるように日射遮蔽膜用塗布液を作製した。この塗布液を用いて実施例18と同様の方法で日射遮蔽ガラスを作製しこれを用いて目的とする合わせガラスを作製した。この合わせガラスの光学特性を表2に併せて示す。

【0071】一方A液とB液にビニル系樹脂としてポリビニルブチラール、可塑剤としてトリエチレングリコールージ-2-エチルブチレートを混合し、ITO濃度が0.18重量%、LaB6濃度が0.0025重量%、ポリビニルブチラール濃度が70重量%となるようにビ 30ニル系樹脂組成物を調製し、実施例1と同様の方法で成形された中間膜を作製した。

【0072】そして前記日射遮蔽ガラスと厚さ2.5mmのフロートガラスとを前記日射遮蔽膜が内側に面するようにして前記中間膜を挟み込んで通常の合わせガラス製造法にしたがって加熱・圧着して、合わせガラスを作製した。この合わせガラスの光学特性を表2に併せて示す。

【0073】作製された合わせガラスについてパンメル値を測定した結果、パンメル値は5であり、板ガラスと中間膜との密着性は十分であった。

【0074】(実施例25)A液とK液を混合し、さらにジアセトンアルコールで希釈して、LaB6 濃度が0.24重量%、SiO2 濃度が1.25重量%となるように日射遮蔽膜用塗布液を作製した。この塗布液15gをスピンコーターで厚さ2.5mmのフロートガラス上に塗布し180℃の電気炉に入れて30分間加熱し、前記フロートガラス上に日射遮蔽膜を形成させ日射遮蔽ガラスを作製した。

【0075】一方A液とB液にピニル系樹脂としてポリピニルブチラール、可塑剤としてトリエチレングリコールージー2ーエチルブチレートを混合し、ITO濃度が0.19重量%、LaB6濃度が0.0025重量%、ポリピニルブチラール濃度が70重量%となるようにピニル系樹脂組成物を調製し、実施例1と同様の方法で成形されたシート状中間膜を作製した。

【0076】そして前記日射遮蔽ガラスと厚さ2.5mmのフロートガラスとにより前記日射遮蔽膜が内側に面するようにして前記中間膜を挟み込んで通常の合わせガラス製造法にしたがって加熱・圧着して、合わせガラスを作製した。この合わせガラスの光学特性を表2に併せて示す。

[0077]

【表2】

	ž	色布法	もの	組斥		光学	特性
	租 類	フ化物 濃度 (%)	ITO 濃度 (%)	ATO 濃度 (%)	SiO ₂ 濃度 (%)	可視光 過率透 (%)	日射透 過率 (%)
実施例18	LaB ₆	0. 045	7. 25	0	2. 5	78	55.7
実施例19	LaB ₆	0.06	4. 33	0	2. 5	78	54. 1
実施例20	LaB ₆	0. 09	3. 84	0	2. 5	78	54. 5
実施例21	LaB ₆	0. 11	3. 09	0	2. 5	78	57. 7
実施例22	LaB ₆	0. 17	1. 38	0	2. 5	7 8	59. 4
実施例23	LaB _{6.}	0. 24	0	0	2. 5	7 8	59.6
実施例24	LaB ₆	0. 023	3. 63	0	1. 25	78	58.0
実施例25	LaB6	0. 12	0	0	1. 25	78	57.9

*実施例18~23は通常合わせガラスに用いられる中間膜を使用し、実施例24、25 は日射遮蔽成分を含有する中間膜を使用した。

【0078】以上の各実施例に示されるように、2枚の板ガラスの間に合わせガラス用中間層が介在されている合わせガラスにおいて、高コストの物理成膜法を用いずに簡便な方法で6ホウ化物微粒子、所望に応じてITO微粒子および/またはATO微粒子を含有する添加液をビニル系樹脂に添加して調製されるビニル系樹脂に添加して調製されるビニル系樹脂に添加して調製されるビニル系樹脂に添加して調製されるビニル系樹脂に添加液にバインダーを添加してなる塗布液を板ガラスの内側に面する面に塗布して日射遮蔽膜を形成し、ついで前記中間膜または従来の中間膜を介在させることにより、日射遮蔽機能を有し、かつ可視光域に高い透過性能を有する日射遮蔽合わせガラスを製造することが可能となった。

[0079]

【発明の効果】以上述べた通り本発明によれば、2枚の板ガラスの間に中間層を介在させてなる日射遮蔽合わせガラスにおいて、6ホウ化物微粒子、および所望に応じ

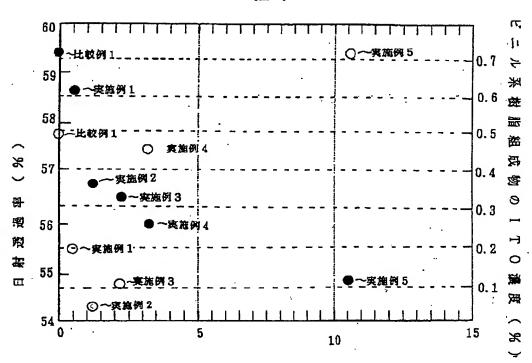
てITO微粒子および/またはATO微粒子を含有する添加液をビニル系樹脂に添加して調製されるビニル系樹脂組成物から形成される中間膜、または前記添加液をバインダーに添加してなる塗布液を板ガラスの内側に面する面に塗布して日射遮蔽膜を形成した後、前記中間膜あるいは従来の中間膜を介在させることによって、日射遮蔽機能を有し、かつ可視光域に高い透過性能を有する日射遮蔽合わせガラスを、高コストの物理成膜法を用いずに簡便な方法で、極めて容易に製造することが可能となった。

【図面の簡単な説明】

【図1】比較例1および実施例1~5におけるITO微粒子の濃度と光学特性の変化を示すグラフ図である。

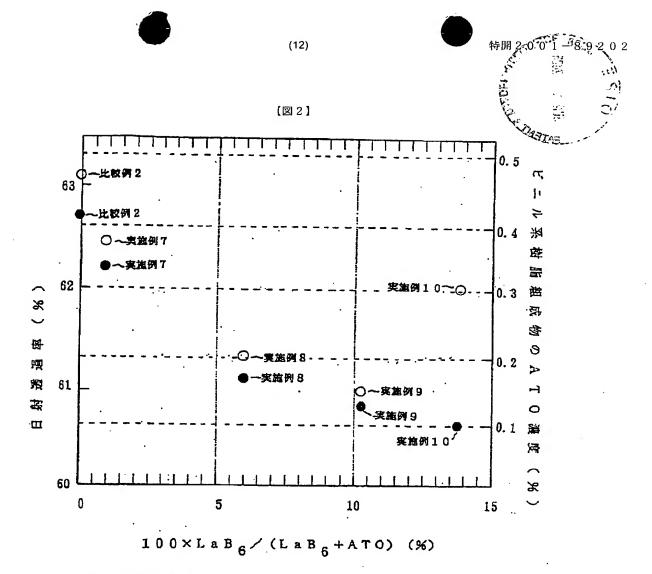
【図2】比較例2および実施例7~10におけるATO 微粒子の濃度と光学特性の変化を示すグラフ図である。





$$100 \times LaB_{6} / (LaB_{6} + ITO)$$
 (%)

- 〇 日射透過率
- ITO濃度



〇 日射透過率

● ·ATO 遊度

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